LA-10563-PR

Progress Report

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Space Nuclear Safety Program

July 1984

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Compiled by
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MASTER

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SPACE NUCLEAR SAFETY PROGRAM

July 1984

Compiled by
T. G. George

ABSTRACT

This technical monthly report covers studies related to the use of $^{238}\text{PuO}_2$ in radioisotope power systems carried out for the Office of Special Nuclear Projects of the US Department of Energy by Los Alamos National Laboratory. Most of the studies discussed here are ongoing; results and conclusions described may change as the work continues.

I. GENERAL-PURPOSE HEAT SOURCE (GPHS)—SAFETY VERIFICATION TEST (SVT) SERIES

A. SVT-6 Postmortem (T. George)

Details of the test conditions and preliminary results were given in previous monthly reports. In this month we received the results of additional fuel analyses and completed the postmortem examination of all test components.

1. Fuel Analyses. Samples for chemical analysis were removed from each of the SVT-6 fuel pellets. The results of wet-chemical analyses for phosphorus are listed in Table I. Significant differences in chemistry were not apparent. The phosphorus content of each fuel pellet was low.

2. Test Summary and Conclusions. On April 5, 1984, the SVT-6 test module was impacted side-on at 54.6 m/s and 975°C. One capsule breached, and all four clads were severely deformed. The breached clad released approximately 0.0149 g of fuel; 0.0061 g of $^{238}\text{PuO}_2$ were released into the catch tube, and 0.0088 g were trapped within the graphite components.

The impact face of capsule HF-373 was breached by two cracks. The cracks apparently resulted from the differential displacement of a large fuel fragment. Both cracks had a brittle, intergranular appearance. Although the breaching cracks traversed coarse-grained (≈ 11 grains/thickness) sections of the vent cup, the shield cup was relatively fine grained (≈ 16 grains/thickness). No

chemical or microstructural anomalies that could be correlated with the wall failures were observed in either cup.

The capsule welds (HF-361 and HF-373) were of acceptable quality. No internal defects were observed during metallographic examination of the weld cross sections.

Examination of the HF-361 and HF-373 vents did not reveal any mechanical defects. Both vents were undamaged and free of glassy oxides or other deposits. However, an unusual amount of grain growth was observed in wall sections adjacent to both vent cover welds. A wall section adjacent to the HF-373 cover weld contained only 2 grains/thickness. Although the abnormal grain growth did not affect impact response, a less favorable impact orientation could have produced extensive clad failures. The occurrence of such pronounced grain growth at the same location in two different capsules suggests a relation to some aspect of the production process.

B. SVT-7 Postmortem (D. Pavone)

The test assembly was aged to simulate an orbital-decay reentry and was impacted with the broad face of the aeroshell oriented at a 15° angle to the target ($\alpha = 15^\circ$, $\beta = \gamma = 0^\circ$). Initial results of the postmortem examination were reported in previous monthly reports. Briefly, major failures occurred on the impact face of capsule FC-364 (primary) and the back side of FC-165 (secondary). A minor failure also occurred in capsule FC-112 (secondary).

Photomicrographs of the FC-364 impact-face failure are shown in Figs. 1 and 2. In both the vent and shield cups, a fuel-fragment indentation was readily apparent. The reduction in thickness of the iridium wall at the fuel-fragment indentation was about 18% in the vent cup and 16% in the shield cup. At the fracture site, a reduction of thickness of about 10% was present in the vent cup, but only a negligible reduction of thickness occurred in the shield cup. A secondary fracture on the interior surface of the shield cup extended through about two-thirds of the wall thickness. A slight reduction in thickness (4-5%) occurred at this location.

Photomicrographs of the failure on the back side of FC-165 are shown in Figs. 3 and 4. The photomicrographs reveal that the iridium deformed at the edge of a fuel-fragment indentation and a fracture in an adjacent region of mild reverse bending. At the point of maximum bending, the shield cup exterior contained small fractures up to about one-third of the wall thickness. The fracture mode was intergranular, with a 4% reduction in thickness near the fracture, and a 12% reduction at the point of maximum bending. Deformation in the vent-cup wall was less severe; essentially no reduction of thickness was detected at the fracture site.

The microstructure of the FC-165 closure weld at the failure site is shown in Fig. 5. The two sides of the fracture do not appear to match, which suggests that some of the weld bead fell out of the sample. The sample sides showed slight radial displacement, indicating that differential fuel-fragment motion imposed nonuniform strains on the capsule wall. The photomicrograph reveals an undesirable microstructure of elongated grains normal to the wall thickness.

The grain sizes of the iridium cups are listed in Table II, and the typical cup microstructures are shown in Fig. 6. The microstructures were generally similar to those observed in previous tests, except that grain size was increased on the interior 20-25% of the wall thicknesses. The grain size of the FC-165 shield cup was significantly finer than that observed in other cups with identical processing histories.

Representative microstructures of the primary capsule closure welds are shown in Fig. 7. The central portion of the FC-364 weld consisted of generally small, nearly equiaxed grains. In contrast, the microstructure of the FC-368 weld contained numerous elongated grains normal to the wall thickness.

Cross sections of the FC-364 and FC-368 vent assemblies are shown in Figs. 8 and 9. Both vent orifices were free of vapor-transported deposits. Only small quantities of plutonia were observed at the entrances to the vent assemblies. Abnormal grain growth was present near both vent assembly and decontamination cover welds.

The microstructures of the HF-364 and HF-368 plutonia fuel pellets were normal (Fig. 10).

Table III presents the results of Auger electron spectroscopy (AES) analysis of samples from fueled clads FC-364, FC-368, and FC-165. Most samples exhibited thorium depletion at the interior surface. Small quantities of phosphorus were detected in a few of the samples. Sulfur appeared in high concentrations in one FC-368 sample and in three FC-165 samples. Extremely high concentrations of carbon and oxygen were detected in one FC-165 vent cup sample; these elements occurred in varying amounts in all of the other samples. The clad failures did not appear to be related to the high concentrations of sulfur, carbon, or oxygen.

Emission spectrographic analysis of samples of the iridium cups showed that iron was the principal impurity. The iron concentration (200 ppm) in these samples was comparable to that observed in samples from previous test clads. The spectrographic results are tabulated in Table IV.

Analyses of the plutonia pellets are summarized in Table I. Although the 300-ppm calcium content of pellet HF-364 exceeded the guideline limit, the concentrations of other impurity elements were well below the guideline limits.

Visual and metallographic observations indicated that fuel-fragment push-through was the primary cause of the major clad failures (FC-364 and FC-165). A high sulfur concentration in the grain boundaries of some samples was the only abnormality observed. Though a major failure occurred in FC-165, no failures were observed in FC-368, which also had high grain-boundary sulfur concentrations. The effect of sulfur segregation to the grain boundaries is not well established.

C. SVT-8 Postmortem (T. George)

Details of the test conditions and the module disassembly were given in the previous monthly reports. In this month we began metallographic examination of the test components, submitted samples of the iridium cups and $^{238}\text{PuO}_2$ fuel pellets for chemical analyses, and received the results of a particle size analysis conducted on the FC-437 fuel.

1. Fuel Analysis. Samples for chemical analysis were removed from each of the SVT-8 fuel pellets. Results of the analyses, presented in Table I, did not show any significant differences in chemistry. Note, however, that the calcium contents of HF-391 and HF-441 were unusually high. HF-391 also had an elevated aluminum content, and HF-437 contained 35 ppm of phosphorus.

2. Particle Size Analysis. To quantify the impact response of the fuel, we selected capsule FC-437 for a particle size analysis. The capsule was opened under water (to prevent the loss of small fuel fragments) and

defueled. The fuel fragments then were sized by passing them through a series of screens. Results of the sieve analyses are given in Tables V and VI.

D. SVT-9 Module Impact Testing (C. Frantz)

The SVT-9 test module was impacted at standard orbital-decay reentry conditions, which were 975°C, 54.57 ± 0.18 m/s, and module orientation of $\alpha = 30^\circ$, $\beta = 0^\circ$, $\gamma = 0^\circ$.

II. SAFETY TECHNOLOGY

A. Iridium Chemistry (K. Axler)

In a recent experiment we attempted to react thorium, iridium, and phosphorus to form a ternary phase. A mixture of thorium, iridium, and phosphorus (in proportions equivalent to a ThIrP₂ ternary compound) was heated to 1400°C for 1 h under vacuum. Subsequent x-ray diffraction revealed trace amounts of

ThP and Th₃P₄ with numerous unidentified lines. A sample of the mixture was sent for quantitative chemical analysis to determine the major components.

We also attempted to form a Th/Ir/P ternary by crystallization from a copper flux. The reactants were combined with copper powder, placed in an Al₂O₃ crucible, and sealed within an evacuated quartz tube. The tube was placed in a computer-controlled furnace and heated to 1250°C, and it is now being slow-cooled to 900°C over a 12-day period.

B. Actinide Phase Studies (K. Axler and D. Peterson)

We attempted to prepare a PuMg sample by melting the appropriate mixture in a tantalum tube sealed under argon. The sample was heated at 1000°C for 5 h. Examination of the product revealed that during the heat treatment the sample wet the tantalum surface and spread across the tube interior; sample recovery was not possible. The experiment will be repeated with a larger sample.

TABLE 1. Plutonia Analyses—GPHS SVT Series

Pellet No.	Selected Elements (ppm)								
	P ^a	Mg	Ca	Al	Si	Fe	Cr	Ni	Ti
SVT-1									
HF-232	5	1	130	20	15	60	15	nd	5
HF-238	5	1	200	25	70	70	15	5	5
HF-261	5	nd ^b	200	20	25	85	40	nd	7
HF-410	2	3	400	25	45	110	35	5	7
SVT-2									
HF-355	4	2	40	25	30	65	30	nd	10
HF-369	3	2	30	15	15	45	10	nd	5
HF-273	3	2	40	25	70	55	25	7	5
HF-449	2	2	40	35	50	80	30	nd	10
SVT-3									
HF-350	3	nd	200	35	20	35	10	nd	20
HF-343	5	10	100	35	30	20	10	nd	15
HF-454	2	nd	200	60	50	70	60	nd	20
HF-457	3	nd	150	35	10	20	15	nd	3
SVT-4									
HF-348	4	3	40	40	10	30	15	nd	7
HF-354	3	3	50	40	15	35	25	nd	7
HF-139	3	2	30	25	8	20	25	nd	10
HF-162	2	2	30	20	8	25	9	nd	5
SVT-5									
HF-260	40 ^c	nd	30	320	15	20	5	nd	15
HF-267	55 ^c	2	40	75	35	30	6	nd	15
HF-415	6	nd	20	40	8	7	nd	nd	15
HF-426	6	nd	30	60	10	10	15	nd	20
SVT-6									
HF-189	3	3	nd	65	25	20	nd	nd	20
HF-225	9	10	60	45	30	15	nd	nd	10
HF-361	8	20	150	30	35	10	15	nd	20
HF-373	2	4	130	15	45	20	nd	nd	20
SVT-7									
HF-364	5	20	300	20	40	5	7	nd	30
HF-368	15	1	25	15	30	nd	10	nd	20
HF-165	9	10	50	30	40	10	15	nd	15
HF-112	8	10	50	30	40	10	15	nd	15
SVT-8									
HF-391	11	7	250	360	35	nd	9	nd	15
HF-436	15	7	nd	50	15	nd	nd	nd	15
HF-437	35	7	6	55	15	9	nd	nd	15
HF-441	6	7	400	75	5	nd	7	nd	10

^aColorimetric analysis.^bNot detected.^cGamma-scan indicates <20 ppm for HF-260 and HF-267.

TABLE II. Iridium Grain Size—GPHS SVT Series

Clad No.	Cup No.	Grains per Nom. 640 μm	Comments
SVT-1			
232	LR313-1	25	Uniform
	LR316-1	21	Uniform except for tab weld
238	LR315-2	20	Uniform
	LR318-6	28	Uniform
261	P709-6	22	Uniform
	Q804-1	22	Uniform
410	S2-5	20	Uniform
SVT-2			
355	Q806-2	21	Uniform
	Q806-5	25	Uniform except for tab weld
369	NR528-2	22	Uniform
	NR528-3	28	Uniform except for tab weld
449	S27-2	23	Uniform
	S25-2	23	Uniform
SVT-3			
343	PR718-5	20	Large internal grains 20 to 25% of thickness
	PR719-5	16	Large internal grains 20 to 25% of thickness
350	R904-7	18	Large internal grains 20 to 25% of thickness
	R904-5	18	Large internal grains 20 to 25% of thickness and tab weld
454	S39-5	15	Large internal grains 20 to 25% of thickness, isolated large external grains
SVT-4			
348	QR820-1	18	Large internal grains 20 to 25% of thickness
	QR820-6	15	Large internal grains 20% of thickness and tab weld
354	QR820-3	19	Large internal grains 20% of thickness
	QR820-4	17	Large internal grains 20% of thickness

TABLE II. (cont)

Clad No.	Cup No.	Grains per Nom. 640 μm	Comments
SVT-5			
267	P701-5	14	Large internal grains 30 to 35% of thickness
	P708-5	16	Large internal grains 20 to 25% of thickness and tab weld
260	PR717-3	15	Large internal grains and isolated large external grains
	PR717-4	16	Large internal grains 20 to 25% of thickness
SVT-6			
361	PR725-5	16	Large internal grains 30% of thickness
	PR726-1	14	Large internal grains 30% of thickness
373	Q803-6	15	Large internal grains 30% of thickness
	PR727-3	16	Large internal grains 30% of thickness
SVT-7			
364	PR726-5	15	Large internal grains 20% of thickness
	PR726-7	16	Large internal grains 25% of thickness
368	PR727-7	15	Large internal grains 25 to 30% of thickness
	NR-529-5	15	Large internal grains 25% of thickness
165	NR-508-4	16	Large internal grains 25% of thickness and isolated large external grains
	NR-507-3	21	Large internal grains 25% thickness

TABLE III. AES Analysis of SVT-7 Iridium Samples

Specimen	Location	Th ₆₅ /Ir ₂₂₉	P ₁₂₀ /Ir ₂₂₉	C ₂₇₀ /Ir ₂₂₉	Os ₅₁₀ /Ir ₂₂₉	S ₁₅₀ /Ir ₂₂₉	Cl ₁₈₀ /Ir ₂₂₉
F-364 Vent cup 1	Interior Edge	0.27	nd ^a	0.43	0.49	nd	0.07
	Center	0.59	nd	0.42	0.77	nd	0.11
	Exterior Edge	0.54	0.06	0.70	1.14	0.07	0.13
	Average	0.47	0.02	0.52	0.80	0.02	0.10
FC-364 Vent cup 2	Interior Edge	0.27	nd	0.23	0.54	nd	nd
	Center	0.53	nd	0.18	0.43	nd	nd
	Exterior Edge	0.42	0.10	0.51	0.89	nd	0.28
	Average	0.41	0.03	0.33	0.63	nd	0.09
FC-364 Shield cup 1	Interior Edge	0.21	nd	0.09	0.40	nd	nd
	Center	0.68	nd	0.13	0.34	nd	nd
	Exterior Edge	0.62	nd	0.20	0.44	nd	nd
	Average	0.50	nd	0.14	0.39	nd	nd
FC-364 Shield cup 2	Interior Edge	0.34	nd	0.32	0.54	0.04	nd
	Center	0.68	nd	0.08	0.30	nd	nd
	Exterior Edge	0.48	nd	0.17	0.20	nd	nd
	Average	0.50	nd	0.19	0.35	0.01	nd
FC-368 Vent cup 2	Interior Edge	0.70	nd	0.13	0.36	nd	nd
	Center	0.84	nd	nd	nd	nd	nd
	Exterior Edge	0.59	nd	0.18	0.31	nd	nd
	Average	0.71	nd	0.10	0.22	nd	nd
FC-368 Vent cup 1	Interior Edge	0.31	nd	0.90	0.78	0.12	0.07
	Center	0.54	nd	0.60	0.98	vs ^b	nd
	Exterior Edge	0.63	nd	0.85	1.18	vs	nd
	Average	0.60	nd	0.78	0.98	vs	0.02
FC-368 Shield cup 1	Interior Edge	0.29	nd	0.15	0.26	nd	nd
	Center	0.85	0.08	nd	nd	nd	nd
	Exterior Edge	0.68	0.13	0.32	0.79	0.04	nd
	Average	0.61	0.07	0.16	0.34	0.01	nd
FC-368 Shield cup 2	Interior Edge	0.19	nd	0.19	0.32	0.05	nd
	Center	0.55	nd	0.17	0.22	nd	nd
	Exterior Edge	0.64	0.12	0.24	0.36	nd	nd
	Average	0.46	0.04	0.20	0.30	0.02	nd
FC-165 Vent cup 1	Interior Edge	0.37	nd	1.20	1.68	vs	nd
	Center	0.53	nd	0.17	0.69	0.07	nd
	Exterior Edge	0.13	nd	0.15	0.47	nd	nd
	Average	0.34	nd	0.51	0.94	nd	nd
FC-165 Vent cup 2	Interior Edge	0.06	nd	4.92	1.34	vs	nd
	Center	0.44	nd	5.75	1.88	vs	nd
	Exterior Edge	0.37	nd	6.38	1.96	vs	nd
	Average	0.29	nd	5.68	1.73	vs	nd
FC-165 Shield cup 1	Interior Edge	0.19	nd	1.08	1.09	vs	0.26
	Center	0.35	nd	0.59	1.30	0.20	nd
	Exterior Edge	0.40	nd	0.56	1.03	0.27	0.10
	Average	0.31	nd	1.08	1.14	vs	0.12
FC-165 Shield cup 2	Interior Edge	0.68	nd	nd	nd	nd	nd
	Center	1.47	nd	nd	nd	nd	nd
	Exterior Edge	0.45	nd	0.24	0.29	nd	nd
	Average	0.87	nd	0.08	0.10	nd	nd

^and—not detected.^bvs—very strong.

TABLE IV. Spectrographic Analyses of GPHS Iridium

Clad No.	Cup No.	Selected Elements ^a (ppm)								
		Cu	Mg	Ca	Al	Si	Cr	Fe	Ni	Pt
SVT-1										
232	LR313-1	3	nd ^b	nd	40	nd	nd	80	10	nd
	LR316-1	20	7	5	50	25	30	150	40	nd
238	LR315-2	15	10	20	50	25	nd	120	40	nd
	LR318-6	15	7	15	50	10	10	120	45	nd
261	P709-6	15	15	20	50	50	nd	150	45	50
	Q804-1	15	15	25	50	30	nd	150	35	150
410	S2-4	3	15	20	50	100	nd	150	50	nd
	S2-5	5	20	30	50	140	nd	120	20	100
SVT-2										
355	Q806-2	nd	3	nd	50	nd	nd	200	30	nd
	Q806-5	10	5	3	60	150	15	200	40	nd
369	NR528-2	3	nd	nd	40	nd	nd	100	nd	nd
	NR528-3	nd	nd	nd	50	nd	nd	150	10	nd
449	S27-2	20	nd	nd	40	nd	10	200	30	nd
	S25-2	10	nd	nd	50	nd	15	200	30	nd
SVT-3										
343	PR718-5	40	nd	nd	40	nd	nd	150	30	nd
	PR719-5	20	nd	nd	50	nd	15	200	60	nd
350	R904-7	6	nd	nd	60	nd	15	300	50	nd
	R904-5	6	nd	nd	60	nd	15	300	40	nd
SVT-4										
348	QR820-1	5	nd	nd	50	nd	10	200	40	nd
	QR820-6	nd	nd	nd	50	nd	10	200	30	nd
354	QR820-3	15	nd	nd	40	10	15	150	30	nd
	QR820-2	40	nd	3	60	10	50	400	80	30
SVT-5										
267	P701-5	15	7	4	50	50	nd	150	60	nd
	P708-5	15	nd	3	60	nd	10	150	50	nd
260	PR717-3	20	nd	nd	60	nd	10	120	50	nd
	PR717-4	20	nd	nd	70	nd	nd	120	50	nd
SVT-6										
361	PR725-5	15	nd	nd	60	10	30	150	40	30
	PR726-1	5	nd	nd	50	nd	15	200	40	nd
373	Q803-6	3	nd	nd	50	nd	40	200	40	nd
	PR727-3	3	nd	3	50	30	30	200	40	nd
SVT-7										
364	PR726-5	15	nd	nd	50	nd	30	200	30	nd
	PR727-5	5	nd	nd	50	nd	20	200	60	50
368	PR727-7	5	nd	nd	50	20	40	200	50	30
	NR529-5	5	nd	nd	50	20	40	200	50	30
165	NR508-4	15	nd	nd	50	18	30	200	30	nd
	NR507-3	15	nd	nd	70	nd	30	200	30	30

^aElements present in excess of the detection limit.^bNot detected.

Table V. Sieve Analysis of the Fuel in Capsule FC-437

Particle Size (μm)	Weight Fraction	Accumulated Weight Fraction
+6000	0.1585	0.1585
+2000	0.4663	0.6248
+ 841	0.2224	0.8472
+ 420	0.0807	0.9279
+ 177	0.0408	0.9687
+ 125	0.0065	0.9752
+ 74	0.0064	0.9816
+ 44	0.0047	0.9864
+ 30	0.0034	0.9898
+ 20	0.0047	0.9945
+ 10	0.0027	0.9972
- 10	0.0028	1.0000

Table VI. Subsieve Particle Distribution of FC-437 Fuel

Particle Size Range (μm)	Weight Fraction	Accumulated Weight Fraction
0 to 1	0.000265	0.000265
1 to 2	0.000144	0.000410
2 to 3	0.000156	0.000566
3 to 4	0.000254	0.000820
4 to 5	0.000135	0.000955
5 to 6	0.000234	0.001189
6 to 7	0.000248	0.001437
7 to 8	0.000185	0.001622
8 to 9	0.000175	0.001797
9 to 10	0.000963	0.002760

(a)

(b)

(c)

Fig. 1. Cross section of the push-through failure in the FC-364 shield cap (a) 6X, (b) and (c) 50X.

(a)

(b)

(c)

Fig. 2. Cross section of the push-through failure in the FC-364 vent cup (a) 6.5X, (b) and (c) 50X.

(a)

(b)

(c)

Fig. 3. Cross section of the push-through failure in the FC-165 shield cup (a) 65X, (b) and (c) 50X

(a)

(b)

(c)

Fig. 4. Cross section of the push-through failure in the FC-165 vent cup. (a) 6.5X, (b) and (c) 50X.

Fig. 5. Microstructure of the FC-163 weld bead at the failure, 50X. Elongated grains normal to the wall thickness are undesirable.

(a)

(b)

(c)

(d)

Fig. 6. Typical microstructures of the SVT-7 iridium cups. (a) FC-364 vent cup, (b) FC-364 shield cup, (c) FC-368 vent cup, (d) FC-368 shield cup, (e) FC-165 vent cup, and (f) FC-165 shield cup. All 100X.

(c)

(d)

Fig. 6. (cont)

(a)

(b)

Fig. 7. Closure welds of the prime fueled clads, (a) TC-364, (b) FC-368. Both at 50X

(a)

(b)

Fig. 8. Cross section of the FC-364 vent assembly. (a) Vent orifice, 50%; (b) vent welds, 50%.

(a)

(b)

Fig. 9, Cross section of the FC-368 vent assembly (a) Vent orifice, 50x, (b) vent welds, 50x

(a)

(b)

(c)

(d)

Fig. 10. Microstructures of the SVT-7 prime plutonia pellets. (a) HF-364, 10x; (b) HF-364, 250x; (c) HF-368, 10x; (d) HF-368, 250x.

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